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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
William J. Mertz, Danny Charles Thompson, Katherine Yiu-Kit Leung
Application No.: 10/657,394
Confirmation No.: 9357
Filed On:: September 8, 2003

Examiner: Margaret G. Moore

Art Unit: 1712

For: RELEASE LINERS AND PROCESS FOR MAKING THE SAME

DECLARATION OF DANNY CHARLES THOMPSON PURSUANT TO 37 CFR 1.132

MAIL STOP RCE
Commissioner For Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Danny Charles Thompson, hereby declare as follows:

1. I am Manager-Product Development with Loparex, LLC, the assignee of the present patent application. Loparex, LLC is the largest commercial supplier of siliconized release papers and films. I am also one of the inventors in the present application.

2. My formal education includes a Master of Chemical Engineering degree from Virginia Tech in 1986.

3. I have over twenty-two (22) years of experience in the field of chemical coatings with emphasis on polymer chemistry and silicone chemistry. As the Manager of Product Development, my primary responsibilities include overseeing new product development projects for specific customer applications.

4. In my present position and throughout my career, I have conducted and supervised research in the development of new products in the silicone release liner area. In addition, I hire, train and supervise other engineers working in the field of silicone release liner research and development. Accordingly, I have a good understanding of the level of skill and knowledge possessed by those of ordinary skill in the art of silicone release liner technology.

5. I reviewed and am familiar with U.S. Patent No. 5,576,356 to Leir et al. The Leir patent is being cited in the present office Action mailed June 12, 2008, to reject claims 1-3 and 5-16 as being anticipated by or in the alternative, obvious in view the Leir patent.

6. I also reviewed and am familiar with the two Eckberg patents, U.S. Patent Nos. 5,258,480 and 5,650,453. These patents are also being cited in the present Office Action to reject claims 1-3 and 5-16 as being anticipated by, or in the alternative, obvious in view of the Eckberg patents.

7. It was previously asserted that the compositions of Leir, which use a reactive diluent is equivalent to the organic solvent in the present invention, and thus results in the release liner having the significantly reduced amounts of total silicone extractables and silicone extractables as in the present invention. It is further asserted in the Office Action that the Eckberg patents teach a radiation curable silicone composition, which is treated with heat and high velocity air, and likewise results in the same product as the present invention. Thus, it is asserted that the cited references inherently are the same product as the present invention.

8. In order to illustrate the differences between the present invention and the compositions of the cited references, I and my laboratory set out experiments to compare examples in the cited references with those of the present application. I recreated the examples in the cited references with equivalent components based on my technical knowledge of one skilled in the art. The following chart lists the corresponding examples in Leir and both Eckberg patents, compared to two examples of products made, one in accordance with Example 6 of the

present application (LO-Ex 1), and a second example corresponding to Example 6 of the present application, but with reduced solids and a different silicone polymer (LO-Ex 2). Units were converted to SI units, where possible.

Loparex Inside and Outside Testing
LO-EX Patent

	Si Coat Wt (grams/sq meter)	Extractable Siloxanes (micrograms/sq cm)	Outgassing Siloxanes (parts per million)	Outgassing Total Organics (parts per million)
Leir Example 33	0.60	2	5.03	57.9
Leir Example 34	0.789	1.86	16.3	116
Eckberg '480 Patent	6.02	1.91	5.7	28.9
Eckberg '453 Patent (A)	0.992	2.02	0.34	16.3
Eckberg '453 Patent (B)	0.667	1.17	0.37	58.9
Loparex LO-EX (1)	0.325	0.42	0.23	0.946
Loparex LO-EX (2)	0.178	0.08	0.74	2.04

A ream is 3000 square feet.

Extractables were measured by the Atomic Absorption method described in the patent application text.

Outgassing testing was done by Innovatech labs according to Seagate Specification #20800020-001. Outgassing was done at 85°C for 3 hours, then the sample was introduced into the GC/MS for analysis.

Components for the experimental formulas and discussion about the coating and curing processes are as follows:

Leir Example 33

GE UV-9300	40 parts
CHVE	20 parts
Limonene Oxide	15 parts
Limonene	25 parts
UV-9390c	3 parts
Total	103 parts

The process by Leir et al. utilized the formula above (col 14, lines 5-12 of the '356 patent), a slot and wire electro-spray process, heat and ultraviolet radiation to cross-link the coating into a durable release surface. Leir considered different temperatures and different line speeds to determine the effect of dwell time and temperature on silicone phase separation and ultimately the release force achieved by the coatings. Coating thicknesses were estimated to be 2.0, 0.7, and 0.4 microns (or roughly 2.0, 0.7, and 0.4 grams/square meter) at the different line speeds. While re-creating the example of Leir, I did not have electrospray process, but used Mayer rods to reach a coat weight of 0.6 grams/square meter on a 50 micron polyester film. A convection oven was used at 160°F for 30 seconds to give the liquid time to flow and mimic the heat cycle of the small oven. UV radiation via medium pressure mercury lamp was used at 1,000 J/m^2 to cure the release formula. Finally, extractables, release tests, and outgassing tests were done to compare the liner produced with this example. Release force with an acrylic adhesive tape was 1.0 N/dm, in line with the results for the 3M acrylic tape, tape B (1.2, 0.7, and 1.0 N/dm), so evidence suggests I reproduced the results of Leir example 33.

Leir Example 34

GE UV 9300	35 parts
CHVE	20 parts
Limonene Oxide	15 parts
Limonene	30 parts
UV-9390c	3 parts
Total	103 parts

Leir Example 34 was made according to the formula described at Col. 15, Lines 52-55. In the example, the solventless release coating was made and coated by an electrospray using a slot and wire technique with a hot air blower as the heat source. As described, air exit temperature was approximately 100°C and web temperature was approximately 50°C. Finally, the coating was passed under a medium pressure mercury lamp at 400 J/m^2. Estimated coating thicknesses are similar to those obtained in Example 33.

I created a version of example 34 using the same coating, with the exception that UV-9390c photocatalyst replaced the now obsolete UV-9310c photocatalyst. Again, Mayer rods were used to generate a coating at a coat weight of 0.789 grams/sq. meter. A convection oven was used to heat the release coated film, again on 50 micron polyester, with an oven temperature of 72°C for 30 seconds dwell. The film sheet was irradiated two passes with a medium pressure mercury UV lamp at 150 W/cm. The cured silicone coating was solid to the touch. The similar battery of coat weights, extractables, release force, and outgassing tests were done and reported above. Coat weight was in the range, and release force was also 1.0 N/dm, similar to the test results reported by Leir. Again, based on these results, I reproduced the example of Leir.

Eckberg '480 patent

UV-9400	100 parts
UV-9390C	3 parts
Total	103 parts

The Eckberg '480 patent uses a rhodium catalyst in the presence of tertiary amine stabilizers, to prevent premature gelling of epoxysilicone materials. The examples describe the synthesis to functionalize the polymer, combining silicone pre-polymers with 4-vinyl cyclohexene oxide (or like moieties) to form epoxy-functional silicones. I did not have access to the UV silicone compositions of Eckberg. However, based on my technical skill and knowledge in the area, I chose GE UV-9400 polymer as an equivalent product to that recited in Eckberg. The 9400 polymer is made using the rhodium complex catalyst with a tertiary amine stabilizer. Furthermore, it goes through a thin film evaporator process (proprietary to Momentive Silicones (formerly GE Silicones)) as described on page 6, lines 17 to 41 of the '480 patent.
The '480 example was generated in the lab using Mayer rods, in this case, a zero rod. The coating was done onto a 50 micron polyester film that had been corona treated prior to silicone coating. The coating was irradiated with UV, two passes at 150 W/cm for each pass in our lab unit. As the application was solventless, the silicone coat weights were higher. I achieved a cured silicone coated release liner. Extractables were 1.9 micrograms/sq. cm. and the total outgassing was 28.9 ppm.

Eckberg '453 patent (A formula)

UV-9400	50 parts
CHVE	50 parts
UV-9390c	3 parts

Total	103 parts

The invention of Eckberg and O'Brien, the '453 patent, is to dilute epoxysilicones with vinyl ether monomers or oligomers to provide UV cured release coating compositions. Silicones and vinyl ether monomers, however, are not soluble, so good mixing practices are essential, and phase separation has to be accounted for in all the coating and curing processes. For example, reducing line speed can cause more of the silicone to separate to the surface, giving lower release force (similar to the Leir patent). Another disadvantage is that atmospheric moisture hinders the cure of the vinyl ethers, significantly more than for epoxyfunctional silicones.
Polymer C from the '453 patent (col. 10, line 62) is a 300 cstk viscosity epoxysilicone fluid. Again, as I did not have access to the actual Eckberg Silicone composition, we chose a commercial silicone, again, UV-9400. The polymer is a multi-functional epoxysilicone, similar to polymer C, with average viscosity of 225 cstk, and again based on my technical skill and knowledge in this area, determined that it would be equivalent to the Eckberg silicone composition. I blended this polymer with CHVE, described as a viscosity reducer and an additive, which adds hardness to the crosslinked coating. In an effort to mimic the approach to diluting the silicone portion to reduce extractables and volatiles, I made the example formula described above as well as the one below, for comparison. By comparing the 453 (Formula A version) to the '480 example, the dilution of the silicone portion was:

- Not effective in reducing silicone extractables (2.02 vs. 1.91 micrograms/sq cm)
- Effective in reducing silicone volatiles (0.34 vs. 5.7 ppm)
- Marginally effective in reducing total volatiles (16 ppm vs. 29 ppm)

Eckberg '453 patent (B formula)

UV-9400	30 parts
CHVE	70 parts
UV-9390c	3 parts
Total	103 parts

The purpose was to take this exercise one step further in diluting a silicone with a vinyl ether, and look at the effect on extractables and outgassing. Here, the silicone extractables were in the 50/50 blend at 2.02 micrograms/sq cm, and in the 30/70 blend at 1.17 micrograms/sq cm. Siloxane outgassing was essentially equivalent (0.34 vs. 0.37 ppm). However, total outgassing was sacrificed in the 30/70 blend (58 ppm vs. 16 ppm for the 50/50 blend). So, this dilution showed an improvement in extractables, but at a sacrifice of total volatiles from the outgassing/GC-MS testing.

LO-EX™ Generation 2 (reduced solids version of Example 6 in the patent application)

PC-601	10 parts
Heptane	135 parts
Isopropanol	40 parts
PC-702	0.25 parts
Total	185.25 parts, 5.4% solids.

In this example, the solvent blend was modified slightly from the heptane/toluene blend we generally use in production, so that we can get better wet-out of the silicone in the lab process. The coating was applied with #4 Mayer rod, then placed in the oven at 250° F for 30 seconds. It was then irradiated with UV energy, two passes with a medium pressure mercury lamp rated at

150 W/cm. Samples were cut and sent for testing. As shown in the results above, actual silicone coat weight was only slightly lower than theoretical at 0.325 gsm (theoretical was 0.35 gsm), with good extractables at 0.42 micrograms/cm^2, and excellent total outgassing at 0.946 ppm. The PC-601 is a good choice for the base polymer, as it is a higher molecular weight polymer than most chosen for UV solventless applications. Release force is easy at 25 grams/inch (0.1 N/dm).

LO-EX™ Generation 3

PC-615	20 parts
Heptane	200 parts
Isopropanol	50 parts
PC-702	0.6 parts
Total	270.6 parts, 7.4% solids

This example represents another embodiment of our invention, this time using a highly functionalized epoxy polymer to produce a release liner with extremely low extractables and low volatiles. The release liner was made in a manner similar to the preceding example, made in our laboratory equipment. Again, Mayer rods were used to meter the coating to the desired silicone coat weight, this time using a #2 rod with the 7.4% solids solution. Actual coat weight is 0.178 gsm, a bit lower than theoretical value of 0.25 gsm. The coated film was placed in the convection oven, which was set at 250° F for 30 seconds. Then, the coating was exposed to UV irradiation, two passes under the medium pressure mercury lamp rated at 150 W/cm. Cure was excellent, as confirmed by the extractables at 0.08 micrograms per square centimeter, and total outgassing at 2.04 ppm. The release force was measured to be 0.2 N/dm, higher than the example above, but lower than some of the earlier examples. The combination of lower molecular weight

polymer and higher crosslink density performed as expected; we achieved lower extractables but slightly higher volatiles than with our generation two product. The extractables were also helped by the lower silicone coat weight.

9. It is acknowledged that in the examples of Eckberg '480, the polymers were prepared by the patent holder. I was not equipped to do polymer synthesis, so I looked for a commercial product made using the processes described in the patent: a rhodium catalyst is used with a tertiary amine stabilizer, and the polymer is processed through a thin film evaporator, the process described in the '480 patent (p.6, lines 18-41). UV-9400 fits those parameters. Second, the results in the table above are given in units as described in the patent application. Extractables are listed in micrograms per square centimeter, and the Volatiles (Outgassing/GC-MS testing) are listed in parts per million of the release liner. Third, concerning the silicone coat weight; in the laboratory examples, efforts were made to reproduce the coat weights as either described in the patents or as would be appropriate for the chemistry selection. The examples made for the Leir patents were appropriate for solventless silicone systems, and match the values given in the patent. The Eckberg '480 patent was a bit more difficult to match, since, the teaching is about making the epoxysilicone polymers. The Eckberg '453 samples were made using solventless processes, slightly below the deposition as described in the patent of 1 g/m^2. Since these coatings are cured with UV energy, Beer's Law applies, so it is not a given that more silicone coating leads to more extractables or volatiles. Energy applied must be absorbed, and sometimes thicker coatings do that more effectively. Therefore, coating weights do not always dictate the levels of extractables or volatiles in the resulting products.

10. As one of ordinary skill in the art, it is my opinion that the compositions of Leir and Eckberg cannot result on the same product with the extremely low levels of total volatiles as in the present invention. This is clear from the data presented above. The examples of the present application illustrated above provide an analysis of the volatile content by outgassing,

wherein the main component is siloxanes. The release liners of the present invention have a 1/10 as much outgassing material as a release liner prepared using the process described in Leir and both Eckberg patents. Thus, neither Leir nor the Eckberg patents provide a release liner with the same low level of volatiles as in the present invention.

11. Similarly, it is also my opinion that the composition of Leir cannot result in the same product with extremely low levels of extractables as the present invention. This is illustrated by a comparison of the data presented above. Similarly, the level of extractables recorded for the Eckberg patents likewise are at levels higher than that of the present invention.

12. Based on the above facts, it is my opinion that neither Leir nor the Eckberg patents disclose, teach or suggest a release liner comprising a radiation curable release coating dissolved in an organic solvent coated onto a surface of a substrate having unexpected and significantly reduced amounts of extractables. Furthermore, it is my opinion that neither Leir nor the Eckberg patents disclose, teach or suggest that the resultant coating has no more than about 1.5 micrograms per square centimeter total extractables and no more than 10 ppm total volatile compounds. The Eckberg '453 (b) coating contains ten times as much total volatiles as does the present invention. There is no experimental evidence in any of the cited references that the level of extractables in their products is the same or less than in the present invention. Therefore, it is not inherent that Leir et al. or either Eckberg patent discloses a product with the same properties, such as low levels of extractables, as in the present invention.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that there statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Respectfully submitted,

Danny C. Thompson

Danny Charles Thompson

Date: December 11, 2008